

## CHAPTER III

### EXPERIMENTAL

In this research, the materials and equipment, experimental and analyzing method were shown below:

#### 3.1 Materials

- Anionic extended surfactant  $[C_{14-15}H_{29-31}-(PO)_8-SO_4Na]$  was supplied by Sasol North America (Lake Charles, LA). This surfactant has a sulfate head group and a hydrophobic tail of methyl-branched alkyl of 14-15 carbons with eight polypropylene oxide (PO) groups having an active content of 29.6 %. The surfactant was used as received from the manufacturer.

- Renewable based nonionic surfactants were sorbitan stearate (SS) and sorbitan oleate (SO). These surfactants were purchased from Sigma-Aldrich.

- Nonionic surfactants were alcohol ethoxylated surfactants. These surfactants were supported by Thai Ethoxylate Co., Ltd (Thailand). Surfactant  $C_{12-14}H_{25-29}-(EO)_n-OH$  series have an alkyl group consisting of a linear hydrocarbon chain with an equal mixture of 12 and 14 carbons, and  $n= 3, 5$  and 9 polyethylene oxide (EO) group, respectively.

- Palm olein was purchased from Morakot Company, as commercial grade.

- Palm stearin was donated by Bangchak Biofuel Co., Ltd. (Thailand).

- Analytical grade sodium chloride (NaCl) was purchased from RCI Labscan with 99 % purity.

- Analytical grade oil soluble dye Oil-red-O was purchased from Sigma-Aldrich.

- D-Limonene was purchased from Chemipan Corporation Co., Ltd.,

### 3.2 Equipment

- 15-mL flat-bottom screw-up tubes
- Micropipette
- Tensiometer (Krüss, K10T)
- Spinning drop video tensionmeter (SVT 15, Dataphysics)
- Incubator (BINDER, KB400/E2)
- Ventilated hood
- Oven
- Water bath
- Shaker
- UV/VIS spectrophotometer (Shimadzu 1800)
- Weighting machine

**Table 3.1** Properties of anionic extended and nonionic surfactants

Surfactants	HC	% Active	MW (g/mol)	HLB Value
Anionic extended surfactant				
$C_{14-15}H_{29-31}-(PO)_8-SO_4Na$	$C_{14-15}$	29.6	715.5 <sup>a</sup>	37.6 <sup>a</sup>
Nonionic surfactants				
$C_{24}H_{46}O_6$			430.6 <sup>b</sup>	4.7 <sup>b</sup>
$C_{24}H_{44}O_6$			428.6 <sup>b</sup>	4.3 <sup>b</sup>
$C_{12-14}H_{25-29}-(EO)_3-OH$	$C_{12-14}$	99.7	332 <sup>c</sup>	8.0 <sup>c</sup>
$C_{12-14}H_{25-29}-(EO)_5-OH$	$C_{12-14}$	99.7	420 <sup>c</sup>	10.5 <sup>c</sup>
$C_{12-14}H_{25-29}-(EO)_9-OH$	$C_{12-14}$	99.7	596 <sup>c</sup>	13.3 <sup>c</sup>

<sup>a</sup>Calculate based on the Davie's group contribution factors (Davies, 1957)

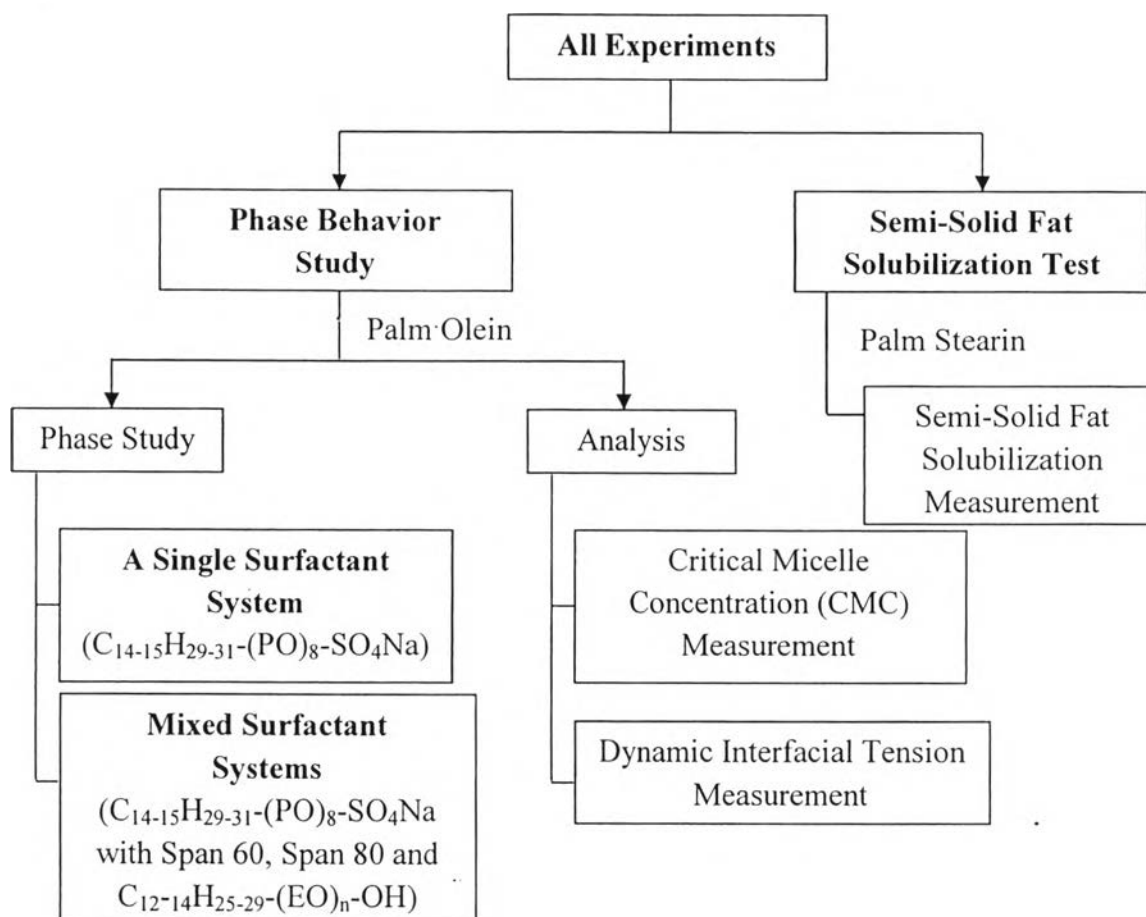
<sup>b</sup>Data from Sigma Aldrich

<sup>c</sup>Calculate based on Giffin's method

HC= number of hydrocarbon chain length

### 3.3 Methodology

#### Overview of Procedure



#### 3.2.1 Microemulsion Formation Experiments

Microemulsion formation experiments divided into 3 parts:

##### 3.2.1.1 *Microemulsion Formation*

Microemulsion formation is performed by adding 5 mL of an aqueous surfactant solution to 15 mL flat-bottom screw-up tubes. The aqueous surfactant solution added was prepared at different surfactant and NaCl concentrations. 5 mL of the palm oleinis then added to obtain an aqueous surfactant solution-to-oil volumetric ratio unity. In this study, both surfactant and NaCl concentrations were prepared based on the aqueous phase. All vials containing the mixtures were shaken gently and placed in an incubator (BINDER, KB400/E2) to

equilibrate for a month at 30 °C. In this work, microemulsion were studied using both single surfactant and with nonionic surfactant systems.

#### *3.2.1.2 Surface Tension Measurement*

The surface tension of surfactant solutions with different surfactant concentrations was determined by surface tensiometer (Krüss, K10T). The surfactant tension measurement was conducted at 30 °C. The transition of plot between surface tension versus total surfactant concentration was used to determine the critical micelle concentration (CMC).

#### *3.2.1.3 Dynamic Interfacial Tension Measurement*

The dynamic interfacial tension (IFT) of the oil and surfactant was determined using a Spinning drop video tensionmeter (SVT 15, dataphysics). The IFT measurements were made using 1-2  $\mu\text{L}$  of oil injected into 300  $\mu\text{L}$  of surfactant solution in a capillary tube. The diameter of the oil drop was measured after 20 min at 30 °C, whereas the rotational velocity was constant.

### 3.2.2 Oil Solubilization Test

#### *3.2.2.1 Dyed Oil Preparation*

Palm stearin (semi-solid fat) was dyed by Oil- Red-O dye using the standard method (Goel, 1998) before being applied on 15 mL flat-bottom screw-up tubes. (100 mL : 0.1 g) and keep in incubator at 60 °C. Approximately 0.1 g of oil-soluble dye having  $\lambda_{\text{max}}$  about 518 nm was added to 100 mL of the oil was prepared for use as color soil for oil solubilization experiments.

#### *3.2.2.2 Cleaning/Solubilization Procedure*

Semi-solid fat solubilization experiments were carried out at a constant temperature of 30 °C. Approximately 0.1 g palm stearin was deposited in 15 mL flat-bottom screw-up tube. Each surfactant concentration was triplication to average oil solubilization values. In order to determine the correlation between phase behavior and solubility, NaCl was added at various concentrations into the tube to obtain the agreement result of IFT result as in the dynamic IFT using spinning drop tensiometer.

### 3.2.2.3 *Semi-Solid Oil Removal/Solubilization Measurements*

Semi-solid fat removal and solubilization were examined by the portion of remaining oil on 15 mL flat-bottom screw-up tube. After 72 hr, semi-solid fat was rinsed gently and dried until remaining weight constant at 75 °C. The semi-solid fat on the glass surface before and after the solubilization experiment was soluted from the tube by d-limonene and the amount of remaining oil in the tube was measured by a UV/VIS spectrophotometer (Shimadzu 1800 liquid) for absorbance at  $\lambda_{\text{max}}=518$  nm. The remaining concentration of oil was calculated from the calibration curve of controlled dye oil solutions.

The solubility of semi-solid fat removal was obtained from the value of semi-solid fat levels on the tube before and after shaking. This method show that the dye and the oil were removed by the surfactant solution in the same proportional which they were loaded on the tube (Goel, 1998).